UNITED STATES DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

SERIAL NUMBER: 09/885,642 ART UNIT: 1762

FILING DATE: 06/20/2001 EXAMINER: Erma C. Cameron

APPLICANT: Kelsoe, Darrell W. DATE OF OFFICE ACTION:

TITLE: Process For Treating Wood and DATE OF RESPONSE: 11/18/2003

Products From Treated Wood

NOTICE OF FILING AFFIDAVITS

The Honorable Commissioner of Patents and Trademarks Washington, D.C. 20231

Sir:

COMES NOW, Darrell Kelsoe, by and through the undersigned counsel and submits to the examiner the attached affidavits of Gopal Nair and Hal Potts as well as the exhibit to Hal Potts' affidavit, his test results for the patentability of the invention disclosed in the referenced patent.

These have been previously submitted in the related '165 case. They show both a superior result and a different process than that.

These show that the prior art fails to achieve a self sustaining exothermic reaction, even when modified to be more closely associated with the invention described herein. They show that because of the failure of the prior art to achieve or suggest a similar result that the invention is novel and is non-obvious.

The prior art must, for obviousness, "both suggest the claimed subject matter and reveal a reasonable expectation of success to one reasonably skilled in the art. In re Vaeck, 947 F.2d 488, 493; 20 USPQ 2d 1438, 1442 (Fed. Cir. 1991)" Ex parte Warne Appeal No. 1997-2509; application no 08/30,982.

Reference is also made to Ex parte Baker, Appeal No. 94-3007 application

07/909,039.

Other authority is available on request.

Respectfully submitted,

GREGORY M. FRIEDLANDER,

Registration No. 31,511

Gregory M. Friedlander & Associates, P.C.

11 South Florida Street

Mobile, Alabama 36606-1934

(251) 470-0303

CERTIFICATE OF MAILING

This message was sent via fax to Erma Cameron, fax number 703-872-9475 on the <u>18</u> day of <u>November</u>, 2003.

GREGORY M. FRIEDLANDER

erial Number: 09/885,642

iling Date: 06/20/01 pplicant: Darrell Kelsoe

itle: Process for Treating Wood nd Products from Treated Wood

Art Unit: 1762

Examiner: Erma Cameron Date of Office ACtion:

Date of Response:

State of Texas County of Harris

Affidavit

My name is Harold Potts.

My technical background is as follows:

BS Science Physics (minor Chemistry) SUNY 1985 New Paltz NY Member Board of Curriculum Advisors Lee College Huntsville Tx

Positions Held Current

Laboratory Manager IRC Inc Houston, Tx Chemical Consultant LCS Labs Houston, Tx

VP Research and Development EMR Inc Houston, Tx 2001-2002 Laboratory Manager Meters & Instruments Inc. Houston Tx 1998-2001 Laboratory Manager Meter Master Inc Houston Tx 1990-1998 Sr Service Planning Engineer Product Development IBM Poughkeepsie, NY 1978-1990

In April of 2003, I performed and completed tests designed in order to compare the disclosures and two Kelsoe patent specifications and in particular with the 09/885,642, filed 6/20/01, with two Saka disclosures 5,652,026, Document Number 8-318,509.

Based on the results which is attached as Exhibit A of those tests, I was able to determine several things which are included in the report.

A summary of my findings are as follows:

- 1. No version of Saka as disclosed or even modified according to the Kelsoe disclosure was able to sustain a detectable exothermic reaction.
- 2. Efficacy of the treatment and the effectiveness of treatment without heat in the Kelsoe invention was significantly and even surprisingly greater than the treatment in either of the Saka disclosures even when modified to be more similar to Kelsoe.

3. As a result of my experimentation, I was able to determine that there was no suggestion in Saka to generate a reaction such as that shown in the Kelsoe document and in fact a careful reading and an experimentation with Saka would not be likely to result in an invention with similar performance to that practiced according to the Kelsoe specification which is a with similar performance to that practiced according to the Kelsoe specification which is a spontaneous on contact with wood, requires no external heat and is capable of treating either green or dried wood products.

In addition, I viewed the following definitions of obviousness:

An obviousness analysis requires that the prior art both suggest the claimed subject matter and reveal a reasonable expectation of success to one reasonably skilled in the art. In re Vaeck, 947 F.2d 488,493,20 USPQ2d 1438 1442 (Fed. Cir. 1991)

(The board attributes to the 'hypothetical person' knowledge of all prior art in the field of the inventor's endeavor....)

Unobviousness is shown where persons having ordinary skill in the art would not have been motived to do what the inventors have done with reasonable expectation of obtaining the invention. The prior art teaching as a whole, given the possibility of and the rewards for success, invention would have to be sufficient to invite skilled artisans to look at compounds the invention would have to be sufficient to invite skilled artisans to look at compounds structurally similar to known agents in the hope of finding new agents. However, obvious to try or obvious to experiment is not the standard for obviousness under 35 U.S.C. § 103. In re or obvious to experiment is not the standard for obviousness under 35 U.S.C. § 103. In re O'Farrell. 853 F.2d 894, 903, 7 USPQ2d 1673, 1681 (Fed. Cir. 1988); In re Dow Chemical Co., O'Farrell. 853 F.2d 894, 903, 7 USPQ2d 1529, 1532 (Fed. Cir. 1988).

Based on my observation and the test results and based on my careful and thorough review of the Kelsoe specification in my review of following patents it is my opinion that Kelsoe is not obvious.

I also had an opportunity to review the two Saka patent documents and also:

Kokai JP8-318509 Meyers 3,682,675 Ogiso 3,682,675

I saw nothing in any of those which would in any way indicate that Saka could be modified in order to produce a Kelsoe type invention nor did any of those suggest a similar invention. In fact, given the long felt and much sought after treatment using similar chemicals, without success, this is an indication of non-obviousness. One with all of the art before him, would not have thought to create an exothermic reaction given the lack of success in that arena.

Further, my experience is in applied science, as opposed to theoretical science. The Kelsoe protocols are so greatly more practical in application and in result, that there is a clear and unmistakable leap from the old technologies to that disclosed in the Kelsoe patents.

Based on this, it is my opinion that Kelsoe is non-obvious under the standards set forth above or under any other known scientific standard and is, in fact a surprising and significant departure from the prior art.

Furthermore affiant sayeth not.

Harold Potts

Sworn to and subscribed to before me the undersigned notary in said state and county on this the 14th day of May

Notary Public

My commission expires:

JIMMY J. INGRAM Notary Public, State of Taxas My Commission Expires November 28, 2004

COMPARISON OF NEW PROTOCOLS OF WOOD TREATMENT

A COMPARISON AND CONTRAST OF PATENT CLAIMS BY SAKA et al AND KELSOE

Table of Contents

INTRODUCTION	1
SECTION 1.0 1.1 Solution Preparation Table 1. Compound Abbreviations 1.1.1 Kelsoe K1-1 Solution K1-2 Solution 1.1.2 Saka Table 2. Compound Molecular mass and specific gravity S2-1 Solution S2-2 Solution S2-4 Solution 1.2 WOOD TREATMENT PROCEDURE 1.3 TESTING and ANALYSIS 1.3.2 LEACHING TEST 1.3.4 Treatment of "Green wood" Saka treated Sample (Designated as S)	2 2 2 2 2 3 4 4 5 6 7 8 9 11 14
SECTION 2.0	2-7
Appendix B	
Appendix C	26

INTRODUCTION

(A) The purpose of this document is to report and illustrate any distinctions between the claims specified within the Saka patents and the claims of the Kelsoe patent. In order to provide this illustration, several experiments will be conducted and the results compared. The experiments will use the formulas exactly as defined in the Saka and Kelsoe patents as well as some slight variants of these formulas. For these experiments five solutions based on formulas from each patent will be used. The solutions chosen will have similar constituent components although different formulas are involved, the protocol will be identical in the application of the solutions. The final product, in this case 1" cubes of pine, having been treated to the different formulations, will then be examined and tested to document the similarities and the differences between Saka's and Kelsoe's processes. The Saka formulas and methods were sourced from two documents. One is U.S Patent 5,652,026 (Modification of Wood 7/29/1997) and a translated document identified as Document No. 8-318509 (Method for Manufacturing a Modified Wooden Material 23/03/1996). The comparisons will be presented in two sections. Section one will involve the Saka Patent 5,652,026. Section two will involve the Document number 8-318509

SECTION 1.0

1.1 Solution Preparation

(a) The solutions will be prepared in adequate quantities and stored in individual flasks until used. Use a graduated cylinder to measure out the reagents used in this solution. The reagents used will be of technical grade, just as would be used in a commercial process. In order to simplify the text, abbreviations will often be used for the longer named chemical compounds as outlined in table 1.

Compound Name	Abbreviation Used
Methyltrimethoxysilane	MTMS
Methyltrichlorosilane	MTCS
Trimethylborate	TMB
Hydrochloric Acid	HCl

Table 1. Compound Abbreviations

1.1.1 Kelsoe

(a) Solutions prepared per Kelsoe's formula are disclosed in the document "Kelsoe CIP2 filed 101702" pg 44 lines 8-13 hereafter referred to as solution K1-1. In a second solution using K1-1 as starting solution, hydrochloric acid will be substituted for methyltrichlorosilane and will be referred to as solution K1-2.

K1-1 Solution

545.5ml methanol

250ml methyltrimethoxysilane

25ml trimethylborate

12.5ml methyltrichlorosilane

Step 1. Add the methanol to an appropriate size flask.

Step 2. Gradually add the methyltrimethoxysilane to the flask while slowly moving the flask in a circular motion to mix the reagents.

***CAUTION!!!! methyltrichlorosilane is harmful, use appropriate safety procedures. Read the MSDS for this chemical before handling.

Step 3. Add the methyltrichlorosilane in the same manner as in step 2. CONTINUED NEXT PAGE

- Step 4. Add the trimethylborate to the flask and stopper the flask then shake well
- Step 5. Place a label identifying the solution in the flask as K1-1
- note. A larger quantity of the K1-1 solution was prepared as it will be used in other experiments outside the scope of this procedure.

K1-2 Solution1

69ml methanol

60ml methyltrimethoxysilane

6ml Trimethylborate

6.4ml Hydrochloric acid (37%)

- Step 1. Add the methanol into an appropriate size flask.
- Step 2. Gradually add the methyltrimethoxysilane to the flask while slowly moving the flask in a circular motion to mix the reagents.
- **CAUTION!!!! hydrochloric acid is dangerous and can produce toxic gas, use appropriate safety procedures. Read the MSDS for this chemical before handling
 - Step 3. Add the hydrochloric acid very slowly in the same manner as in step 2.
 - Step.4. Add the trimethylborate to the flask and stopper the flask then shake well
 - Step 5. Place a label identifying the solution in the flask as K1-2

1.1.2 Saka

- (a) Solutions are prepared based on Saka's formula as disclosed in the document "U.S. Patent 5,652,026 Saka et al" page 4 section 6 under the heading "EXAMPLE 2" hereafter referred to solution S2. According to descriptions contained within "example 2" after mixing the reagents and heating for sixty minutes, Saka states "The reaction solution was ripened for one hour at 60° C, obtaining a 65% methanol solution of the oligomer." After reading this, I must reason that Dr Saka is indicating a 65% oligomer component and a 35% methanol component. I have neither the time or resources to scientifically verify this For the purposes of my experiment I will maintain the assumption of a 65% oligomer/35% methanol mix. This assumption will allow the calculation of the amount of methanol required to dilute the "ripened" solution to the 10% concentration as specified in Saka's "example 2".
- (b) The quantities in Saka's artwork are given in grams of reagents. For uniformity of the procedures to follow, the gram equivalents have been converted to milliliters. The procedure used to convert quantities expressed in grams to quantities expressed in milliliters uses the following formula:

Quantity (ml) = Quantity(gr) / Specific Gravity

Compound	Molecular Mass	Specific Gravity	
MTMS	136.22gr	0.95	
MTCS	149.49gr	1.275	
TMB	103.91gr	0.92	
HCl	36.46gr	1.19	
Methanol	32.04gr	0.79	
Water	18.02gr	1.00	

Table 2. Compound Molecular mass and specific gravity

S2-1 Solution²

- 129.5ml methyltrimethoxysilane
- 11.3ml trimethylborate
- 15.0ml distilled water
- 856.9ml methanol
- Step 1. Add the methyltrimethoxysilane into an appropriate size flask.
- Step 2. Add the trimethylborate into the flask.
- Step 3. Place the flask on a magnetic stirring hotplate, heat the solution to 30 degrees C while stirring.
- Step 4. Using a burette, add the distilled water, drop wise until all of the water is in the flask.
- Step 5. Increase the temperature of the solution to 60 degrees C for 60 minutes.
- Step 6. Dilute the solution with methanol to obtain 10% oligimer solution
- Step 7.Place a label on the flask identifying the solution in the flask as S2-1

² See Appendix A for the calculation of the reagent quantities used

S2-2 Solution³

- 129,5ml methyltrimethoxysilane
- 15.0ml distilled water
- 794.8ml methanol
- Step 1. Add the methyltrimethoxysilane into an appropriate size flask.
- Step 2. Place the flask on a magnetic stirring hotplate. Heat the solution to 30 degrees C while stirring.
- Step 3. Using a burette, add the water, drop wise until all of the water is in the flask.
- Step 4. Increase the temperature of the solution to 60 degrees C for 60 minutes.
- Step 5. Dilute the solution with methanol to obtain 10% oligimer solution
- Step 6. Place a label on the flask identifying the solution in the flask as S2-2.

³ See Appendix B for the calculation of the reagent quantities used

SMK Solution4

- (a) To see what was suggested by a combination of Saka and Kelsoe, I took Saka's experiment, applied Kelsoe's technology to see if Saka could be changed to perform like Kelsoe.
 - 30.0ml methyltrimethoxysilane
 - 2.6ml trimethylborate
 - 3.5ml distilled water
 - 1.5ml hydrochloric acid
 - 325.0ml methanol
 - Step 1. Add the methyltrimethoxysilane into an appropriate size flask.
 - Step 2. Add the trimethylborate into the flask.
 - Step 3. Using a burette, add the distilled water, drop wise until all of the water has been added..
 - **CAUTION!!!! hydrochloric acid is dangerous and can produce toxic gas, use appropriate safety procedures. Read the MSDS for this chemical before handling
 - Step 4. Using a burette, add the hydrochloric acid, drop wise until all of the acid has been added..
 - Step 5. Dilute the solution with methanol to obtain 10% oligimer solution
 - Step 6. Place a label on the flask identifying the solution in the flask as S2-4

⁴ See Appendix C for the calculation of the reagent quantities used

1.2 WOOD TREATMENT PROCEDURE

- (a) The treatment procedures to be followed in this document are disclosed in the document "Kelsoe CIP2 filed 101702" pg 44 lines 22-25 and continued on page 45 lines 1-8. Only 1" cubes of seasoned yellow pine wood will be used. The same procedure will be performed using each of the five solutions previously prepared . The procedures will be performed at room temperature and at ambient atmospheric pressure and humidity. A total of four samples will be treated per solution.
- Step1. Gather five 600ml beakers and label each of the beakers with a label indicating K1-1,K1-2,S2-1,S2-2 and SMK.
 - Step 2. Pour 200 -400 ml of each prepared solution into the corresponding beakers
 - Step 3. Gather 20 yellow pine 1" cubes. Using a pencil, mark four of the wood cubes with as follows:

Qty 4 labeled K1-1-1 thru K1-1-4

Qty 4 labeled S2-1-1 thru S2-1-4

Qty 4 labeled S2-2-1 thru S2-2-4

Qty 4 labeled K1-2-1 thru K1-2-4

Qty 4 labeled SMK-1 thru SMK-4

- Step 4. Weigh each cube and record the weight and associated marking to identify the cube. The data is to be recorded on the data sheet in appendix A of this document.
- Step 5. Place five wooden cubes in the beakers ensuring the markings on the cubes correspond to the labels on the beakers.
- Step 6. Submerge using stainless steel weights on top of the wood cubes to prevent them from floating.
- Step 7. Cover the beakers with cellophane and use a rubber band to secure the covering. Allow the samples to soak for 15 minutes.
- Step 8. Remove the treated wood cubes from the beakers and place on a drying rack.

 Allow the wooden cubes to dry for 8 hours.
- Step 9. Weigh each cube and record the weight and associated marking to identify the cube. The data is to be recorded on the data sheet in Table 3.



1.3 TESTING and ANALYSIS

(a) The following tests shall be performed in order to compare the treated wood characteristics brought about by the different solutions. The tests are:

Weight gain

Leaching

Elemental Analysis

(b) The data collected in the procedures section regarding the weight of the wooden cubes provides the data necessary to establish weight gain. The elemental analysis will be performed by an outside laboratory to document the silicon and boron levels contained within the treated wood cubes. In this section, only the procedures for testing of weight gain and leaching will be presented.

1.3.1 Post Treatment Weight Gain Results

Sample ID	Initial Wgt gr	Final Wgt gr	Wgt Delta gr	% Change
K1-1-1	7.9563	8.5292	0.5729	+7.2
	8.4530	9.0251	0.5721	+6.8
K1-1-2	8.3675	8.9431	0.5756	+6.9
K1-1-3	7.5414	8.1119	0.5705	+7.6
K1-1-4	7.5 (2.)			AVG +7.1
G2 1 1	8.3158	8.6802	0.3644	+4.4
S2-1-1	8.5358	8.8875	0.3517	+4.1
S2-1-2	7.4707	7.8065	0.3358	+4.5
S2-1-3	7.8371	8.1998	0.3627	+4.6
S2-1-4	7.0371			AVG +4.4
	7.6977	8.0146	0.3169	+4.1
S2-2-1	8.0174	8.3793	0.3619	+4.5
S2-2-2	_	8.0228	0.3433	+4.5
S2-2-3	7.6795	8.1854	0.3328	+4.2
S2-2-4	7.8526	0,100		AVG +4.3
	7.8668	8.4215	0.5547	+7.1
K1-2-1		8.8450	0.5163	+6.2
K1-2-2	8.3287	8.2820	0.5173	+6.7
K1-2-3	7.7647	8.2847	0.5175	+6.7
K1-2-4	7.7672	0.2047		AVG+6.7
	# ODE4	8.1525	0.1671	+2.1
SMK-1	7.9854	7.7705	0.1576	+2.1
SMIK-2	7.6129	7.9625	0.1561	+2.0
SMIK-3	7.8064	7.9025	0.2501	
SMK-4	7.9424	8.1197	0.1773	+2.2
			1	AVG +2.1

Table 3. Sample weights and post treatment weight gains

1.3.2 LEACHING TEST

- Step 1. Prepare five 600ml beakers each filled with 250ml of distilled water. Label each of the five beakers with the appropriate sample ID.
- Step 2. Place a cube into each of the five beakers while taking care to match the markings on the cube with the beaker label. Use a stainless steel weight placed on the top of the wood cubes in order to keep the cubes submerged.
- Step 3. Cover the tops of the beakers with cellophane and secure the cellophane with a rubber band. Allow the wood to soak for 24 hours.
- Step 5. Remove the cubes from the beakers and softly towel dry each of the samples.

 Weigh and record the weight at 15 minute intervals. This data illustrates the water absorbency characteristics of the wood samples as well as give a general indication of the rate of water absorption of the sample. The test data is shown in Table 4 and the final weight gains are graphed in figure 1.
- Step 6. Drain the beakers into 300ml flasks. As each beaker is drained, label the flask with same identification as the beaker. Stopper the flask, it will be used to determine the extent of treatment leaching if any.
- Step 7. Deliver the water samples to an independent laboratory for elemental analysis.
- Step 8. Record the results in Table5.

Sample	Initial Wgt	15 min.	30 min.	45 min.	60 min.	Wgt Gain	% Gain
K1-2 K2-1 S2-1-3 S2-2-4 SMK-3 Control	grams 8.77 8.70 7.61 7.99 7.86 12.5	grams 8.97 8.88 9.36 8.62 8.33 14.72	grams 9.08 8,98 9.82 9.01 8.75 15.56	grams 9.14 9.05 10.04 9.36 9.09 15.96	grams 9.22 9.12 10.13 9.64 9.51 16.24	grams 0.45 0.42 2.52 1.65 1.65 3.74	% 5.13 4.83 33.1 20.7 21.0 29.9

min.= minutes, wgt = weight

Table 4. Hydrophobic test data

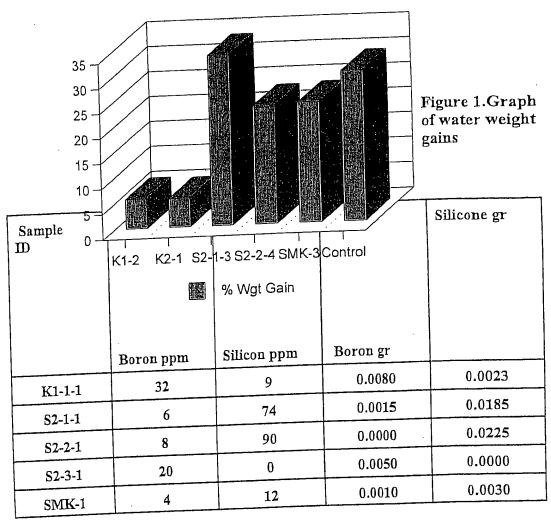


Table 5. Leaching results (leachate present in 250mL of distilled water)

1.3.3 Rates of Water Absorption

(a) The average rates of absorption for each sample are calculated from the test data in Table 2 by using the following mathematic formula:

(Final weight) - (initial weight) / 60 minutes = mL/ min (1gr water = 1mL)

K1-2 0.0085mL/min					
K2-1 0.0082mL/min					
S2-1-3	0.0430mL/min				
S2-2-4	0.0313mL/min				
	SMK-3 0.0312mL/min				
Control	0.0253mL/min				

Table 6. Average Rates of water absorption

1.3.4 Treatment of "Green wood"

(a) To demonstrate the action of the Kelsoe and Saka solutions on green or unseasoned wood a treatment was performed using a formula from Saka and a formula from Kelsoe. The wood samples were taken from a yellow pine tree and were of a branch. From this cut branch two samples were cut approximately 1-1/2" long and approximately 1-1/4" in diameter. The bark was removed from each of them and then both were rinsed off with ethanol. The samples were allowed to dry for 5 minutes before treatment. The time between the cutting of the branch from the tree and treatment was less than 20 minutes. Furthermore a small hole was drilled into the side of each sample. This hole will have a temperature sensor placed within it to measure the temperature during treatment.

Conditions Temperature 24.5° C Pressure 1 atmosphere

Saka treated Sample (Designated as S)

Treatment Formula used: S2-1

Weight Results

Sample weight (pre-treatment): 40.13 gr

Sample weight (post treatment): 40.67 gr

Weight Gain: 0.54 gr

% Weight Gain: 1.36%

Temperature Results

Time Minutes	0	. 5	10	15	20	25	30
Time Minutes	24.5	244	24.4	24.1	23.9	23.4	23.0
Temp Degrees C°	24,5	2-1.1			l	l	

Table 7. Internal Temperature of Sample During Saka Treatment

Conditions Temperature 24.5° C Pressure 1 atmosphere

Kelsoe treated Sample (Designated as K)

Treatment Formula used: K1

Weight Results

Sample weight (pre-treatment): 41.15 gr

Sample weight (post treatment): 40.70 gr

Weight Gain: - 0.45 gr

% Weight Gain: - 1..09%

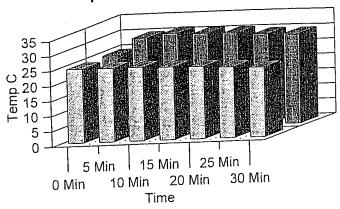
Temperature Results

•							
Time Minutes	.0	5	10	15	20	25	30
I IIIIc Iviinateos	 		20.0	21.7	30.8	30.0	29.9
Temp Degrees C°	24.5	24.5	30.0	31.2	50.0	30.0	

Table 8. Internal Temperature of Sample During Kelsoe Treatment

Kelsoe's formulations heated during treatment.

Temperature vs Time



Kelsoe Saka

Figure 2. Time vs

Temperature Comparison of Saka and Kelsoe

Section 1 Observations and Conclusions

GENERAL

- (a) The preparation of the five solutions produced no extraordinary observations, all were clear with the exception of Saka based solution S2-1. The solution S2-1 had a slight milky appearance that turned clear upon heating at 60° C, an indication of a reaction proceeding when heated.
- (b) During the treatment of the wood samples, small streams of bubbles could be observed escaping from the ends (longitudinal with the grain) of all five samples. The rate and volume of the gas bubbles leaving the wood was considerably small. The samples were of seasoned dry wood and most likely the source of the gas bubbles was escaping air from the wood as it was displaced by the solution, but since the gas was not collected and analyzed no definitive conclusion can be reached.

Weight gain:

- (c) The Kelsoe formula and it's variant (K1-1 and K1-2) showed a significantly greater sample weight gain after treatment than did any of the Saka based formulas (refer to Table 3, (30% or greater). The difference in weight gains produced by Kelsoe's formulas versus those of Saka's formulas is most likely the result of the Kelsoe formula consisting of, for the most part, an un-reacted mixture of reagents, that has a dependency on substances within the wood to initiate a reaction.
- (d) Conversely, Saka's formula, in effect, reacted the solution before it was introduced into the wood. This distinction is of importance when contrasting the protocols of Saka versus Kelsoe. In the Saka Patent document referenced in this paper, Saka teaches us that in the preparation of his solutions, oligomers are produced. These oligomers are of larger molecular structures and have higher molecular weights than the monomeric precursive compounds. The larger molecules would experience a higher resistance in diffusing into the wood than the smaller monomers. This is likely the reason Saka calls for a relatively high dilution factor. Therefore it could be reasoned that for a unit of time of treatment, less oligomer of Saka's is introduced inside the wood structures than smaller monomeric compounds of Kelsoe under conditions specified by Kelsoe. This explanation appears to be supported by the experimental evidence of the weight gains of the samples and the extraordinary methods of treatment outlined by Saka in his patent (i.e.heat treatment under vacuum and pressure).

Silicon and Boron content:

(e) The amount of silicon and boron introduced into the treated wood samples, as found by an independent lab, was surprising and did not coincide with my expectations. All the levels of boron and silicon were found to be much lower than anticipated. The average level of boron in the sample treated by the two Kelsoe formulas was 14.5ppm and the average silicon level was 3.5ppm. The Kelsoe samples had a range of boron levels from 2ppm to 2ppm. The samples treated with the Saka formulas resulted in in an average level of boron at 3ppm and an average of 4.7ppm of silicon. The ranges of boron and silicon respectively were 1ppm to 6ppm and 2ppm to 8 ppm. The sample designated S2-2-2 show the presence of boron. This is problematic due to the intended absence of any boron containing compounds in the S2-2 treatment solution. There are several

possibilities that could account for this discrepancy, the glassware or utensils had been contaminated with a boron compound during solution preparation, the non-boron samples made contact with the borated samples during transport or handling, or the sample was contaminated at the independent lab during analysis. There are currently plans to perform the procedure a second time ensuring additional precautions are taken to prevent any cross contamination.

(f) Referring to Table 5, it's shown the Saka formulas resulted in a slight increase in silicon placement into the wood samples compared to the Kelsoe formulas. In contrast the Kelsoe formulas resulted in a significantly higher level of boron placement over the Saka variants.

Leaching of Si and Bs

- (g) The leaching test data also indicates a major contrast between the formulas of Saka and Kelsoe. A greater amount of silicon escaped from the Saka treated samples than those of Kelsoe (refer to Appendix B). According to Saka's procedure the treated wood needs to be heated to complete the treatment process. This would indicate a secondary reaction is required to "fix" the silicon in the wood.
- (h) Since the experiments outlined in this paper were performed in accordance with the Kelsoe protocol, with no heat treatment of the samples, the secondary reaction did not initiate or did not approach completion. Therefore the silicon contained in Saka's formula would not have been able to "fix" into the wood structures and thus be highly susceptible to leaching by way water immersion as was indicated by the leaching test data.
- (i) Kelsoe had significantly less leaching of silicon. The Kelsoe formula reacts within the wood as hydrolysis occurs with the water content as well as possibly reacting with the cellulose by replacement reaction or cross-linking. In either instance the test data shows that significantly higher levels of silicon remain inside the wood. It is important to note that the level of silicon retention of the Kelsoe protocols are achieved at ambient temperature and pressure.
- (j) The leach test data also shows that samples treated had distinct differences in boron retention. [The Saka formula showed low levels of boron leaching. This is likely due to the extremely low boron content. Conversely the Kelsoe formula leached on average more boron, but in this likely due to the greater boron content found in the treated samples.
- (k) The data reflecting the silicon-boron impregnation and the leaching of same shows a contradictory finding. The amounts of boron and silicon leached out into water were vastly greater than the amounts found in the samples not soaked in water. Some possible explanations could be that the samples used in the leaching tests were not washed before being immersed in water, so that the silicon and boron existed on the surface of the wood in greater concentrations than inside the wood and this surface boron washed off into the water. Conversely, the samples that underwent elemental analysis were washed during the preparation of the samples prior to pyrolysis. The original samples used in the leaching tests are currently being prepared for another run of the same tests to discover if the amount of leaching changes, since in effect all of the leached samples have been washed during the initial test.

Water Absorbing:

M

- (I) Kelsoe shows a water resistance 5 (five) times that of Saka. The test data shows overwhelmingly the superiority of the Kelsoe formulas over those of Saka relative to water absorbency. The Kelsoe formulas provided a level of water resistance up to five times better than Saka's and six times that of untreated wood. The sample used as the control was larger than the treated samples, but was taken from the same board. The calculation of percentage of weight gain is not affected by the size of the sample. The sample S2-1-3 showed a higher percentage increase than the control, as of this moment, the reason is not clear.
- (m) It has been observed the rate of a liquid into a porous substance exhibits similar rates of change per the time intervals. The rate reduces by approximately half in each time interval. The test data presented here follows that approximation and thus behaved as normal.

Conclusion:

(n) Based on these preliminary findings it appears that the protocols of Saka and Kelsoe are similar only in the precursor reagents used in the treatment solutions. The protocols diverge from form and function beyond that point. It appears to be obvious that the commercialization of the Kelsoe protocol would be significantly more viable than the protocol of Saka due to the extraordinary steps required to prepare the wood and the post treatment step (heating and vacuum application).

Green Wood Treatment: Temperature: Kelsoe's generates heat and is therefore exothermic:

- (o) The treatment of "green wood" was performed to compare the performances of the Kelsoe and Saka formulas. The results showed some striking differences. The plotting of internal temperature versus time clearly indicates an exothermic reaction taking place with the Kelsoe formula as indicated by the temperature increasing initially, then stabilizing, and finally a decrease. But, examining the weight gain measurements of the Kelsoe sample a loss in weight is shown. This fact coupled with an apparent exothermic reaction might seem contradictory.
- (p) Before this sample was treated, noticeable amounts of resinous sap was present at the ends where it had been cut with a saw . During treatment, these ends still leaking sap developed a dark purple color that covered the entire cut ends. This purple coloring diffused throughout the solution as time passed. The coloring was most likely the production of an ion complex(ion complexes are usually brightly colored).

Hypothesis:

(q) The reaction that caused the coloring is yet unknown as is the nature of the ion complex, but it appear to involve the sap and one or more of the compound within the solution. The fact the purple color traveled into the solution indicates movement of a substance from within the wood and out the ends into the solution. Thus is it possible more substance escaped from the sample than entered. The temperature rise can be more easily explained. The temperature probe was placed into a drilled hole that was located on the middle of the sample on the circumference. There was ample clearance between the probe and the sides of the hole. This clearance allowed treatment solution to enter and actually react with the surface area of wood. The volume within the hole is small, but the proximity of the probe and the insulating quality of the wood would enable the limited volume involved in the reaction to be detected by the probe. The sample treated by Saka



behaved in an opposite fashion, no noticeable heat, but a slight weight gain. There was not any discoloration of the solution during the Saka treatment. The weight gain was about one fourth of the expected weight increase given the performance of this solution with seasoned wood. This is probably because Saka relies on absorbtion (not reaction) to get reagents into the wood for subsequent treatment.

I also used dye in experiments with Kelsoe and Saka. After exposing those to wood. I cut slices from the samples. The finding showed a more deep purple (the color of the dye) and a darker purple in the Kelsoe sample than the Saka sample. A comparison with water confirmed that Saka's comparison was observationally similar to chemicals being absorbed. Kelsoe is deeper and more through dye indicated the dye was being carried onto the wood by the reaction.



SECTION 2.0

(a) This section will deal the Saka formula disclosed in the Saka Document number 8-318509 modified to be more similar to the Kelsoe formula. This is no longer Saka, this is now a hybrid of Kelsoe and Saka. The focus of this section will be on the effects of a strong acid acting as a catalyst, in this instance, hydrochloric acid as disclosed in Kelsoe. Two separate solutions were prepared in which to treat the wood samples. The only difference between the two was the addition of HCl (to act as a catalyst). The two solutions were made up of the following:

Saka w/ acetic acid and w/o HCl (Designated 1S)

1.4mL methyltrimethoxysilane (0.01M)

103.9mL trimethyl borate (1.0M)

58.2ml ethanol (1.0M)

0.6ml acetic acid (0.01M)

*note: per the Kelsoe protocol no heat was applied the solution

Saka w/ acetic acid and w/ HCl (Designated 2S) MK - Saka method to be more similar to Kelsoe

1.4mL methyltrimethoxysilane (0.01M)

103.9mL trimethyl borate (1.0M)

58.2ml ethanol (1.0M)

0.6ml acetic acid (0.01M)

4.4mL hydrochloric acid

*note: per the Kelsoe protocol no heat was applied the solution

(b) The samples were treated for 30 minutes each at ambient pressure and temperature. The results of the treatments follows:

Conditions: ambient temp 26.7° C pressure 1 atm

1S solution

				5	10	15	20	25	30
Time	1	2	3			050		25.0	24.9
Temp	25.6	25.7	25.6	25.5	25.3	25.2	25.1	23.0	
ļ <u>-</u>	2.0	3.0	3.8	4.0	5.2	5.3	5.3	5.3	5.3
Press	2.0	3.0	3.0				·		

Time is elapsed treatment time in minutes

Temp is temperature in degrees C

Press is the internal pressure of the wood in unit of 1/8 inches of water column

2S solution

			·						
m:	1	2	3	5	10	15	20	25	30 :
Time	I					05.77	25.5	25.2	25.2
Temp	25.8	26.0	26.0	26.0	25.8	25.7	25.5	23.2	23.2
Tomp				2.0	2.0	2.0	2.0	2.0	2.0
Press	1.2	1.5	1.8	2.0	2.0	2.0	1	L	<u> </u>

Time is elapsed treatment time in minutes

Temp is temperature in degrees C

Press is the internal pressure of the wood in unit of 1/8 inches of water column

Hydrophobicity Tests

(a) The air dried samples were immersed in beakers containing 250mL of distilled water. The samples were kept in the water for 4 hours upon which they were removed, towel dried and weighed. These samples will be weighed repeatedly over the next 24 hours to track the weight changes. In addition to weight gain, the conductivity of the water will be measured to detect changes in the conductivity of the distilled water. The conductivity is proportional to the amount of ions or other charge carriers in the water. Thus the higher the conductivity, the more ions(most likely from the boron and/or chlorides) have escaped the treated wood. While not a specific count of the ions, the conductivity will give an excellent relative indication of leaching.

Sample 1S Results

Weight

r only)

The Mho is a unit of conductance and is defined as the reciprocal of the resistance (ohm).

Weight

Initial weight before immersion	13.12 gr
Post Immersion weight (<5 mins)	15.39 gr
· Weight change	2.27 gr
% Weight change	17.3%
Post Immersion weight (8 hrs)	12.91 gr
Weight change (8 hrs)	- 0.21 gr
% Weight change (8 hrs)	-1.60%
Conductivity	
Initial Conductivity	1.946 uMho (Distilled water only)
Final Conductivity	24.390 uMho
% Change	1181.1%

The Mho is a unit of conductance and is defined as the reciprocal of the resistance (ohm).

Section 2 Observations and Conclusions

- (a) Saka did not work, even with an acid catalyst, to generate an exothermic reaction.
- (b) No visible changes in any of the solutions (ie color or turbidity) were observed. The solutions during mixing, experienced no enthalpy changes as I could detect by monitoring the temperature. The results of the 1S and 2S MK solutions indicate that neither treatments were effective in providing water resistance or even noticeable weight gains after treatment. Neither generated an exothermic reaction. The 2S MK solution had an acid catalyst added to increase the hydrolysis of the TMB in oxides. After studying the results it is obvious there was minimal reactions, hydrolysis or otherwise and the acid did not contribute any detectable catalytic effect. The 1S and 2S MK samples only had significant weight gains when measure immediately after leaving the treatment solution. Given the weights after the samples had dried for a few hours show that the weight gains were apparently from the alcohol in the solution saturating the wood. The small quantities of acetic acid and trimethyltrimethoxysilane involved would not lead to significant reactions or subsequent products. The apparent weight loss experienced by sample 2S MK was in part caused by efflorescence of either the boron or a chloride containing compound as was observed as white "fuzzy crystalline" growths emanating from the surface of the sample. Theses structures were so fine as to break off and fall away from the wood and are not counted as weight. There is also the likelihood the alcohol dissolved some of the resins or gums contained within the wood into the solution or the white crystalline formations.
- (c) The internal pressure was measured in these tests. The method of this measurement uses a water manometer connected to a short length of small diameter glass tubing. The glass tube was then tightly inserted into a hole of the same diameter making a tight seal. The principle relies on Boyle's Law. The gases contained within the wood are slightly compressed as solution enters the wood. The placement of the tube places it in the approximate center of mass equidistant from either end of the sample. The rate of the pressure increase would therefore be in proportion to the rate of penetration. The pressure stabilizes when there is no further transit of solution into the wood, indicating that perhaps the wood is saturated. The pressure level can indicate two conditions, the amount of material penetrating the wood, and the other is the quantity of gas trapped in the wood (more gas filled voids in the wood). Greater gas content contained within the wood, would indicate a reduced moisture content. The 1S treatment showed higher pressure during treatment and the cause is still under study. The pressures of both samples showed similar points where the pressure stabilized, indicating the samples were saturated at approximately 3-5 minutes.

HYPOTHESIS

(d) The reaction is the boron compound breaking down to Boric Acid. No significant silicon hydrolysis occurred in the work sample. The borate will react with water, whether the acid is there or not.

CONCLUSION

(e) Saka does not suggest or teach a method of using hydrochloric acid to conduct an



exothermic reaction.

Appendix A

S2-1 Solution Calculations

129.5ml methyltrimethoxysilane

11.3ml trimethylborate

15.0ml distilled water

856.9ml methanol

(a) The first step in determining the quantity of methanol is find the total amount of the reagents,

$$129.5$$
ml + 11.3 ml + 15.0 ml = 155.8 ml

(b) This is the total amount of solution that is heated and according to Saka produces 65% oligimer and 35 % methanol. Using this proportion then,

total oligomer (ml) =
$$0.65 \times 155.8 \text{ ml} = 101.27 \text{ml}$$

total insitu methanol(ml) = $0.35 \times 155.8 \text{ml} = 54.53 \text{ml}$

(c) Since the goal is to obtain a 10% solution of oligomer and the amount of oligomer we have is 101.27ml, this must already represent 10% of the final solution. Thus the remaining 90% would be 9 x 10% or 9 x 101.27ml which is 911.43ml. This means the amount of methanol necessary in a 10% solution of oligomer would be 911.43ml. Now since according to Saka's artwork we started with 54.53ml of methanol created by the reaction. We therefor subtract 54.53ml from the 90% amount to arrive at the amount that must be added,

$$911.43 - 54.53$$
ml = 856.90 ml methanol

Note1: The actual solution prepared had all quantities listed reduced by 50% Note2: TMB was figured into the equation even though it is uncertain of the contribution to oligomer reaction.

Appendix B

S2-2 Solution Calculation

129.5ml methyltrimethoxysilane

15.0ml distilled water

794.8ml methanol

(a) The first step in determining the quantity of methanol is find the total amount of the reagents,

$$129.5$$
ml + 15.0 ml = 144.5 ml

(b) This is the total amount of solution that is heated and according to Saka produces 65% oligimer and 35 % methanol. Using this proportion then,

total oligomer (ml) =
$$0.65 \times 144.5 \text{ ml} = 93.93 \text{ml}$$

total insitu methanol(ml) = $0.35 \times 144.5 \text{ml} = 50.58 \text{ml}$

(c) Since the goal is to obtain a 10% solution of oligomer and the amount of oligomer we have is 101.27ml, this must already represent 10% of the final solution. Thus the remaining 90% would be 9 x 10% or 9 x 93.93ml which is 845.37ml. This means the amount of methanol necessary in a 10% solution of oligomer would be 845.37ml. Now since according to Saka's artwork we started with 50.58ml of methanol created by the reaction. We therefor subtract 50.58ml from the 90% amount to arrive at the amount that must be added,

$$845.37 - 50.58$$
ml = 794.79 ml methanol

Note: The actual solution prepared had all quantities listed reduced by 50%

Appendix C

SMK Solution Calculation

30.0ml methyltrimethoxysilane

2.6ml trimethylborate

3.5ml distilled water

1.5ml hydrochloric acid (37%)

325.0ml methanol

(a) This solution had to be prepared in a lesser quantity due to a diminished supply of MTMS. To ensure proper proportions of reagents in this solutions, the percentage of reduction will calculated. To achieve this, merely the find the percentage of available reagent to the original quantity,

$$30\text{ml} / 129.5\text{ml} = 0.232$$

$$\% = 0.232 \times 100 = 23.2\%$$

(b) This percentage represents the value all components used in this solution needs to be reduced in order to maintain equivalency. Using this percentage the following quantities are found,

TMB
$$11.3$$
ml x $0.232 = 2.62$ ml

Water
$$15.0$$
ml x $0.232 = 3.48$ ml

(c) The HCl that was added as a catalyst, is intended to be at a 1.5% concentration relative to the oligomer component. So adding the oligomer components,

$$30.0$$
ml + 2.6 ml + 3.5 ml = 36.1 ml

$$36.1 \times 0.015 = 0.542$$
ml

(d) The HCl used is a 37% solution and we intend to correct for a 100% solution. This is accomplished by increasing the quantity of HCl solution to yield 0.542ml of pure HCl. This is performed by the following:

$$0.542$$
ml $/ 0.37 = 1.46$ ml

Roron nnm	Silicon ppm	Boron gr	Silicon gr
	9	0.0080	0.0023
6	74	0.0015	0.0185
8	90	0.0000	0,0225
20	0	0.0050	0.0000
4	12	0.0010	0.0030
	Boron ppm 32 6 8 20 4	32 9 6 74 8 90 20 0	32 9 0.0080 6 74 0.0015 8 90 0.0000 20 0 0.0050

Table 5. Leaching results (leachate present in 250 mL of distilled water.

Serial Number: 09/885,642

Filing Date: 06/20/01

Applicant: Darrell Kelsoe

Title: Process For Treating Wood and

Products From Treated Wood

Art Unit: 1762

Examiner: Erma Cameron Date of Office Action:

Date of Response:

I, M. Gopal Nair, hereby declare as follows:

BACKGROUND

I currently serve as Professor of Biochemistry and Molecular Biology at the University of South Alabama College of Medicine. I am a Senior Scientist and I am also the Director of the Drug Development Laboratory at the University of South Alabama, Department of Biochemistry and a Scientist at the Comprehensive Cancer Center at the University of Alabama in Birmingham.

I have served as Chairman of the Department of Biochemistry and Molecular Biology of the college of Medicine for two years beginning 1990, and then served as Vice-Chairman through September 2001. I have received numerous Awards from National Cancer Institute, American Cancer Society and American Heart Association for the past two decades to pursue my drug development efforts. I have advised Private corporations, including BioNumerik Pharmaceuticals, Inc. on the procedures of writing Investigational New Drug(IND) applications, FDA regulations, conflict of Interest, compound manufacture and formulations and served as a consultant to a number of private industries. My professional piers are at the highest levels of outstanding and dedicated scientists who are leaders in their respective fields and work at various prestigious Universities in the United States and throughout the world.

I have also served as Major Professor, Mentor, Director and Guide to the Ph.D. students throughout their career and tenure in my laboratory. Further, I have taught, directed and guided the research of a number of post-doctoral research associates who

were hired by me to carry out chemical research in the areas of compound synthesis and formulation. I have more than 160 publications. My contributions in compound synthesis and formulations are widely recognized nationally and internationally.

I am Inventor or co-Inventor of several Patents. All of my patents are new inventions developed in my laboratory as potential treatments for human diseases. I have been invited several times by Federal Government Agencies to serve as a member on committees on drug development for cancer, AIDS and other diseases.

I have served Fossil Rock as a paid consultant during the year 2001 and I was personally involved in supervising and studying the properties of the formulations described in the claims in the instant patent application. This expert report is written for a fee on an hourly basis that will be billed to Fossil Rock for payment at a rate of not more than \$200.hr.

I have had extensive practical and theoretical experience with protection and deprotection of amino, hydroxyl and carboxyl groups of complex organic molecules by silylation and in phosphorous chemistry and hydroboration.

This experience is relevant in the analysis of the patentability of the application 09/885,642 because this application deals with silicon and boron chemistry with emphasis placed on the reaction of hydroxyl groups of various wood molecules. Further it deals with the novelty, chemical differences and advantages of the present invention as opposed to the cited, but not relied up on prior art

I have experience in Patent filing and prosecution in the United States and world-wide. I have personally drafted applications (including claims) for a number of United States and international Patent applications. The prosecution of each of my Patent applications resulted

in allowable claims and issued Patents. For further details regarding my experience, please see my curriculum vitae which is attached for the Examiner's convenience.

ANALYSIS OF KELSOE PATENT

Collectively the above knowledge and experience have provided me with the required expertise to comment on the Patentability of the claims of the >642 application over the cited but not relied up on prior art documents. I have personal knowledge of wood treatment invention of the >642 application and have personally evaluated the theoretical mechanism by which the reactions take place.

The invention does provide simultaneous hydrophobicity, fire retardancy and microbial protection to wood treated by Applicant's invention.

DISTINGUISING THE CLAIMS OF THE >642 APPLICATION

Distinguishing Over Document JP 8-318509 To Saka

plicant's Invention Is
shed Over JP 8-318509
genated alkylsilanes in
nineral acid formation
ts wood treatment
s distinctly different and
ed in composition and
e .
į

Distinguishing Over JP 8-318509 To Saka In View Of US 3,682,675 To Meyers

Applicant's Claimed	Disclosure Of Saka	Disclosure Of	Claimed Elements not	Why Applicant's Invention Is
Element	(P 8-318509)	Meyers	disclosed	Distinguished Over Saka (JP 8-
į į				318509) In View of Meyers
Pro-catalyst in the	No pro-catalyst in	No pro-catalyst in		No similarity in the treatment
formula	the treatment	the treatment formula that come		formula that comes in contact with
·	formula	formula		wood.

THIS PAGE 5 (FIVE) IS INTENTIONALLY LEFT BLANK

5

Distinguishing Over JP 8-318509 To Saka In View Of The Disclosure Of Ogiso

			Cit I de la Flancata pot	Why Applicant's Invention Is	
Applicant's Claimed	Disclosure Of JP 8-	Disclosure Of Ogiso	Claimed Elements not	why Applicant's invention is	
Element	318509 by Saka		disclosed	Distinguished Over Saka In View	
				of Ogiso	
The formula consists	No halogenated	Uses SOL-GEL		The applicant's formula is	
of the crucial	alkylsilane in the	method with		designed on the inclusion of a	
halogenated mono,	formula	ultrasound		halogenated mono, di or	
di or trialkylsilane.		Sol-Gel is an		trialkylsilane in an otherwise well	
		oligomer of ill		defined stable chemical formula.	
		defined composition			
	} 	. Does not have any		For this single reason alone the	
		halogenated pro-		applicant's formula is novel and	
		catalyst in the		unexpected.	
		treatment formula			

Distinguishing Over JP 8-318509 To Saka In View Of US 5,652,026 To Saka

Applicant's Claimed	Disclosure Of Saka in JP 8-	Disclosure Of Saka	Claimed Elements	Why Applicant's
Element	318509		Not Disclosed	Invention Is
				Distinguished Over
				Saka
Halogenated mono, di	None	Mixtures of oligomers		There are no
and trialkylsilanes as		of boron, silicon and		similarities of the
pro-catalysts		phosphorous of ill-		instant formula with
		defined composition		the oligomer gels of
		<u> </u>	<u> </u>	<u> </u>

	Saka
SAKA(U.S. 5,652,026)	KELSOE APPLICATION
Saka Formula is entirely different. It has no relation to	Hydrophilic solvent (ethanol) is used
the Kelsoe's formula. The formulae are mixtures of polymers made outside	Completely homogenous and stable composition that is crystal clear and extremely compatible with all kind of wood and wood products.
the wood in a separate chemical process. This formula has no resemblance or similarity with the Kelsoe's	and extremely companione with all kind of wood and wood products.
composition at all.	
	Mixes completely with water
Wood needs to be CONDITIONED prior to treatment with unrelated composition. Acetone extraction with a southlet for several hours should precede water	Will penetrate both wet and dry wood
conditioning of wood to desired moisture level. This is a time consuming and expensive procedure.	No conditioning of wood is required.
	No evolution of any gases. Environmentally safe.
	Does contribute to anti-microbe protection by boron
	Forms non-leachable boron/silicon matrix on all parts of treated wood.
	Simultaneous microbial, water and fire resistance are obtained by the
	Kelsoe's composition.
	Formula contains a mixture of three components: An activatable silicon
	additive, an activatable boron reagent and a pro-catalyst that on contact

	with wood molecules activates the silicon additive and boron reagent	
	simultaneously and exothermically to form stable covalent bonds of	
	boron and silicon atoms with wood molecules containing hydroxyl groups.	
SAKA et al. JP2962191(1999); 08318509 (1996)		
Needs hydrolysis, thermal decomposition and poly-	1. No vacuum required	
condensation of the formula to be partially effective.	2. No heating of any amount required	
Even after these three discreet steps applied to wood	3. The reaction is spontaneous and exothermic	
after treatment 6% of metal oxide is leached out	4. The pro-catalyst in the formula facilitates covalent bond	
indicating that the metal is NOT covalently attached	formation between hydroxyl groups of wood molecules and the	
to wood molecules.	metal atoms (boron and silicon) to form permanent bonds.	
1. The wood piece needs to be in contact with	5. No heaching of metal takes place	
formula under vacuum (15 mmHg) for three	6. The presence of the pro-catalyst in the instant patent formula	
days.	aids spontaneous reaction of wood hydroxyl groups with metal	
2. Needs heating at 65 degree centigrade for 24	atoms without the need of. Hydrolysis, pyrolysis and poly-	
hours (endothermic process; heat must be	condensation all highly endothermic processes required by the	
provided) to decompose.	teachings of Saka Patent 08318509.	
3. Then heating required for 24 hours at 105		
degrees. A highly endothermic reaction for		
poly-condensation.		
SCHULDT et al. 1995:14754		
A laboratory process to protect hydroxyl groups of		
cellulose with trimethylsilyl group is described. The		
technology does not teach the use of a mixture of boron		
and silicon compounds to impart hydrophobicity	This well-known laboratory procedure is not significant to the commercial	
microbial resistance and fire retardancy to wood o	technology of this instant patent application.	
wood products. The technology is not relevant to th	e	

In U.S. Patent 5,652,026 to Saka et al., the disclosure technology treats wood with a methylsiloxane oligomer which contains phosphorous and/or boron. These are high molecular weight co-polymers that need to be prepared separately from trialkoxysilanes and other reagents by heating and diluting. The material to be applied on to wood is not an alkyltrialkoxysilane but a pre-prepared copolymer. Saka does not disclose Applicant's claimed composition.

The wood to be treated by the Saka formula is moisture conditioned first by soxhlet extraction with acetone, a very expensive procedure followed by re-exposure to moisture to the desired water content. This conditioned wood is then dipped for three days in the oligomer formula in methanol under vacuum. Essentially the oligomer is forced to be drawn in to the pores of wood by mechanically applied vacuum and the methanol is simultaneously removed. The wood then undergoes heat treatment at 65 degrees for one day and then at 105 degrees for another day requiring costly fuel expenditure. This wood had a weight gain of 11.3%; however on exposure to water for four hours more than one half of the silicon (7.5%) was leached out indicating that the silicon is not bonded to the wood in spite of prolonged and expensive curing of the wood at high temperatures after treatment.

Saka's technology of using the silicon oligomers followed by curing the wood at high temperatures to obtain fire retardancy does not teach the Fossil Rock technology described in the Kelsoe application.

MECHANISM OF REACTION OF KELSOE'S FORMULATION WITH WOOD vs. SOL-GEL PROCEDURE OF SAKA:

Applicant's >642 application is unexpected, new and novel. For example if one evaluate the make-up of the reacting formula of the sol-gel technology with wood vs. the reacting formula of the >642 application there are no similarities.

Saka's disclosure is a mixture of un-reactive gel comprising of oligomers made soluble in an aqueous medium (SOL-GEL formula).

Saka's treatment is un-reactive on contact with wood.

Saka's disclosed technology is based upon SOL-GEL technology. The term SOL-GEL refers to:

- Making a gel like substance of undefined chemical composition by hydrolyzing a mixture of boron, silicon and phosphorous reagents with water. The product thus formed is a mixed oligomer, contained in a reaction vessel outside the vicinity of any wood to be treated.
- The above gel is then made soluble in an aqueous organic solvent. This solution no longer contains or have any similarities with the reagents used as wood treatments in the instant patent.
- The wood to be treated is separately extracted with acetone in a special complex apparatus known as the soxhlet to remove water. The wood is then dried and re-exposed to moisture to get the desired moisture content for the wood to be treated.
- 4) The wood is then placed in a chamber and vacuum is applied for

approximately three days to evacuate the wood.

- The evacuated wood while in vacuum is then exposed to the solubilized oligomer gel formula to have the formula enter the wood. The wood is subsequently removed after forced impregnation of the sol-gel under vacuum and then dried at 60 degrees.
- 6) No reaction has yet taken place among wood molecules and the oligomer formula.
- 7. The wood is then heated at 105 degrees for another 24 hours to make the reaction work at least partially. Both silicon and boron leaches out of the wood in significant amounts on exposure to water after pyrolysis.
- 8. SOL-GEL stands for Soluble Gel. The composition of the formula consists of oligomers of polysiloxanes prepared from hydrolysable silicon, boron and phosphorous compounds that are treated with water and hydrolyzed to oligomers of unknown composition.
- 9. This mixtures of oligomers are then dissolved in methanol/water to make the formula to treat wood. Saka utilizes a mixture of oligomers in aqueous methanol prepared outside wood prior to treatment. This formula is not related to Applicant's invention.

Saka's oligomers whether outside or inside the wood are unable to react with wood molecules unless activated by heat for prolonged periods (24 hours at 60 degrees and another 24 hors at 105 degrees. In order for the formula to penetrate wood of 1.0 mm thickness(!) a vacuum must be applied for three days. In addition the wood piece must be preconditioned

with soxhlet extraction and then moisture conditioned. The entire procedure is so cumbersome and expensive that in all probability will not result in profitable industrial application.

In contrast, the >642 application has a well-defined chemical formula that is completely homogeneous, reproducible and stable. No expensive and time consuming (soxhlet extraction with acetone, moisture conditioning, applying vacuum and pyrolysis) preconditioning of wood is needed for the reactions to occur. The formula is absorbed to ALL parts of wood by simple contact (dipping, brushing or spraying). No application of vacuum is necessary. Once absorbed and in contact with wood molecules having hydroxyl groups (cellulose, lignins etc.) the pro-catalyst present in Kelsoe's patent formula releases hydrogen chloride in catalytic amount that in turn activates the silicon and boron additives for reaction with hydroxyl groups of wood molecules resulting in covalent bond formation. These reactions are spontaneous and exothermic as opposed to the highly endothermic conditions required for the SAKA technology. Once the catalyst is released on contact with hydroxyl groups of wood molecules, the Kelsoe formula is self-activated to initiate a chain reaction that eventually results in the covalent bonding of both boron and silicon to wood molecules. Therefore the boron and silicon are not leached out in any significant amounts as experimentally verified in the specification section of the Kelsoe application.

The Saka method that relies on the Sol-Gel procedure is of theoretical interest but it is useless as a profitable and viable method for wood treatment in the lumber industry. Saka method teaches impregnation of a gel prepared separately outside the wood, by hydrolyzing a hydrolysable silicon compound with or without addition of hydrolysable phosphorous or

boron compound to form un-reactive mixed gels of undefined composition. This gel is then dissolved again outside the wood to be treated in a mixture of methanol water. Therefore the Saka formula is a solution of an un-reactive mixed gel of unknown composition. The Kelsoe formula is a crystal clear solution the composition of which is well defined and which on contact with wet or dry wood results in spontaneous and instant mild exothermic reaction to render wood simultaneous hydrophobicity, fire retardancy and microbial resistance.

Applicant's claimed invention is a stable solution of well defined composition consisting of a pro-catalyst and a catalytically activatable silicon reagent with or without an appropriate boron additive. A pro-catalyst is a compound that on demand is converted to a catalyst. The demand in the present invention is exposure to hydroxyl groups (or moisture within wood) of various wood molecules on contact. A catalytically activatable reagent is a reagent that is un-reactive unless activated by the catalyst. In the present invention a mineral acid is produced *in situ* on instant contact with wood molecule that in turn activates the boron and silicon additives for instant reaction with wood molecules to form covalent bonds.

One pro-catalyst used in the instant formula is methyltrichlorosilane that on contact with hydroxyl groups will generate a strong mineral acid that in turn will activate the silicon and boron additives for instant covalent bond formation with wood molecules via the hydroxyl oxygen. No where in the cited up on prior art there is a technology with the instant formation of a catalyst from a pro-catalyst that in turn will activate un-reactive silicon and boron additives for instant reaction with wood molecule on contact to provide hydrophobicity, fire retardancy and microbial resistance. Therefore, this expert respectfully submits that the Kelsoe technology is new, novel and unexpected and is patentable over the prior art teachings.

Kelsoe technology is distinct and different from the Saka procedure which requires evacuation of conditioned wood for three days at 15mm/Hg, Applicant's treatment formula penetrates both wet and dry wood on contact (dipping, spraying or brushing) and instantly reacts with wood to impart the desired properties. For these reasons alone this expert respectfully submit that the Kelsoe composition to treat wood is patentable over the sol-gel procedures of Saka.

Applicant's invention imparts hydrophobicity, fire-retardancy and microbial resistance to both wet and dry wood just by contact. This unprecedented property is built in to the formula by careful theoretical design and selection of pro-catalyst and silicon and boron additives. Once the formula comes in contact with wood molecules having hydroxyl groups the pro-catalyst instantly reacts with hydroxyl groups generating a mineral acid exothermically that protonates the alkoxyl groups of the silicon additive and boron additive. The mineral acid does not escape the wood as gas because of its relative small amounts and the large amount of additives to be protonated. Protonation of the alkoxyl groups of the additives activate them for instant reaction with wood hydroxyl molecules that results on covalent bond formation of silicon and boron atoms with the oxygen atoms of the wood permanently.

The entire wood is thus treated permanently in ONE STEP; JUST CONTACT! Therefore from a mechanistic point alone the technology is novel, new and unprecedented. The composition claims recited in the Kelsoe application are considered patentable over the cited documents taught in SAKA patents.

Kelsoe technology has responded to various drawbacks of previous silicon and boron based technologies for potential commercial wood treatment. None of the cited prior art

PAGE 14

technologies disclose all of the elements claimed by the Applicant, and they do not teach or suggest Applicant's claimed invention (discussed in detail below). Further, the cited technologies have not achieved commercial success and are unlikely to achieve commercial success due to the expensive and time consuming processes and procedures involved. On the other hand the >642 application is simple, convenient, cheap, new, novel and unexpected.

>642 application differs entirely from the above cited documents technology with respect to the composition in the following ways:

Kelsoe composition is chemically well defined and identified.

Kelsoe composition does not make use of aqueous solutions, anhydrous organic solvent is required for the composition. Kelsoe's composition, in one embodiment, utilizes a halogenated silane component as a pro-catalyst. [The concept of pro-catalyst is novel and unexpected in wood industry].

Kelsoe's composition enters wood without prior conditioning or application of vacuum for several days.

Kelsoe's composition instantly reacts with wood hydroxyl groups on contact and activates the accompanying reagents to form silicon-oxygen covalent bonds not only on the surface but also within the wood.

Kelsoe's composition requires no prior drying of wood or no drying of wood after treatment to be effective.

Various Kelsoe's formulations described herein are unique with respect to defined and pure ingredients and the formula must have a halogenated silane and a non-aqueous organic solvent to be effective. None of the cited documents describes the use of a halogenated silane as a pro-catalyst

to activate additives for instant reaction within the wood with wood molecules to form stable covalent bonds.

For these reasons and others the technology described in the instant application is novel, unprecedented and unexpected from SAKA teachings. Therefore, I respectfully submit that the technology described in the Kelsoe application is patentable over the cited but not relied up on prior art documents of Saka and others.

I have reviewed the report of the experiments performed by Potts comparing Saka to Kelsoe.

I found these experiments to be completely consistent with my expectations and my experimentation with the Kelsoe formulations. The results verified the exothermic and spontaneous nature of the reaction with wood accomplished by the invention of the Kelsoe technologies.

Furthermore affiant sayeth not.

M G Nair Ph D

Professor

Sworn to and subscribed to before me the undersigned notary in said state and county

on this the $\frac{19}{2}$ day of

16

MARKED UP PARAGRAPHS

On page 24 lines 18-22, here is the paragraph marked up.

The basic structure of [the] \underline{a} molecule used in the process described herein $\underline{i}\underline{s}$ [include]: \underline{R} - $\underline{X}_{\underline{a}}$ - $\underline{X}\underline{b}_{\underline{3}}$ or \underline{R} - $\underline{R}_{\underline{3}}$ - $\underline{X}\underline{a}$ - $\underline{X}\underline{b}$ where \underline{R} is a carbon compound, $\underline{X}_{\underline{a}}$ is a tetravalent atom (which may be substituted with trivalent or pentavalent atoms and other embodiments), $\underline{X}\underline{b}$ is \underline{a} [\underline{R} is a carbon compound, $\underline{X}\underline{a}$ is a trivalent, tetravalent or penta valent atom $\underline{a}\underline{n}\underline{d}$ $\underline{X}\underline{b}$ is a reactive seven valence] halogen (fluorine, chlorine, bromine, etc.) or their equivalent. On page 28 lines 3-4 here is the paragraph marked up:

SOLVENT: The solvent can vary tremendously also although it is preferably a non water based solvent so as not to cause a reaction or minimal water compound [so it is not going to cause it would typically be structured so as not to effect the glue or other properties of wood composites].